Laboratory Processing Learning Objectives

Learn how to:

- Match process options to analytes and data objectives
- Manage sample moisture
- Select/reduce particle size
- Collect subsamples for analysis
- Apply Quality Assurance
- Examine options for lab certification

Report Documentation Page

Form Approved
OMB No. 0704-0188

1. REPORT DATE	2. REPORT TYPE	3. DATES COVERED
MAR 2012		**00-00-2012 to 00-00-2012**

4. TITLE AND SUBTITLE	5a. CONTRACT NUMBER
Laboratory Processing Learning Objectives	5b. GRANT NUMBER
	5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S)	5d. PROJECT NUMBER
	5e. TASK NUMBER
	5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) **Interstate Technology and Regulatory Council (ITRC),50 F Street NW Ste 350,Washington,DC,20001**	8. PERFORMING ORGANIZATION REPORT NUMBER
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	10. SPONSOR/MONITOR'S ACRONYM(S)
	11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited

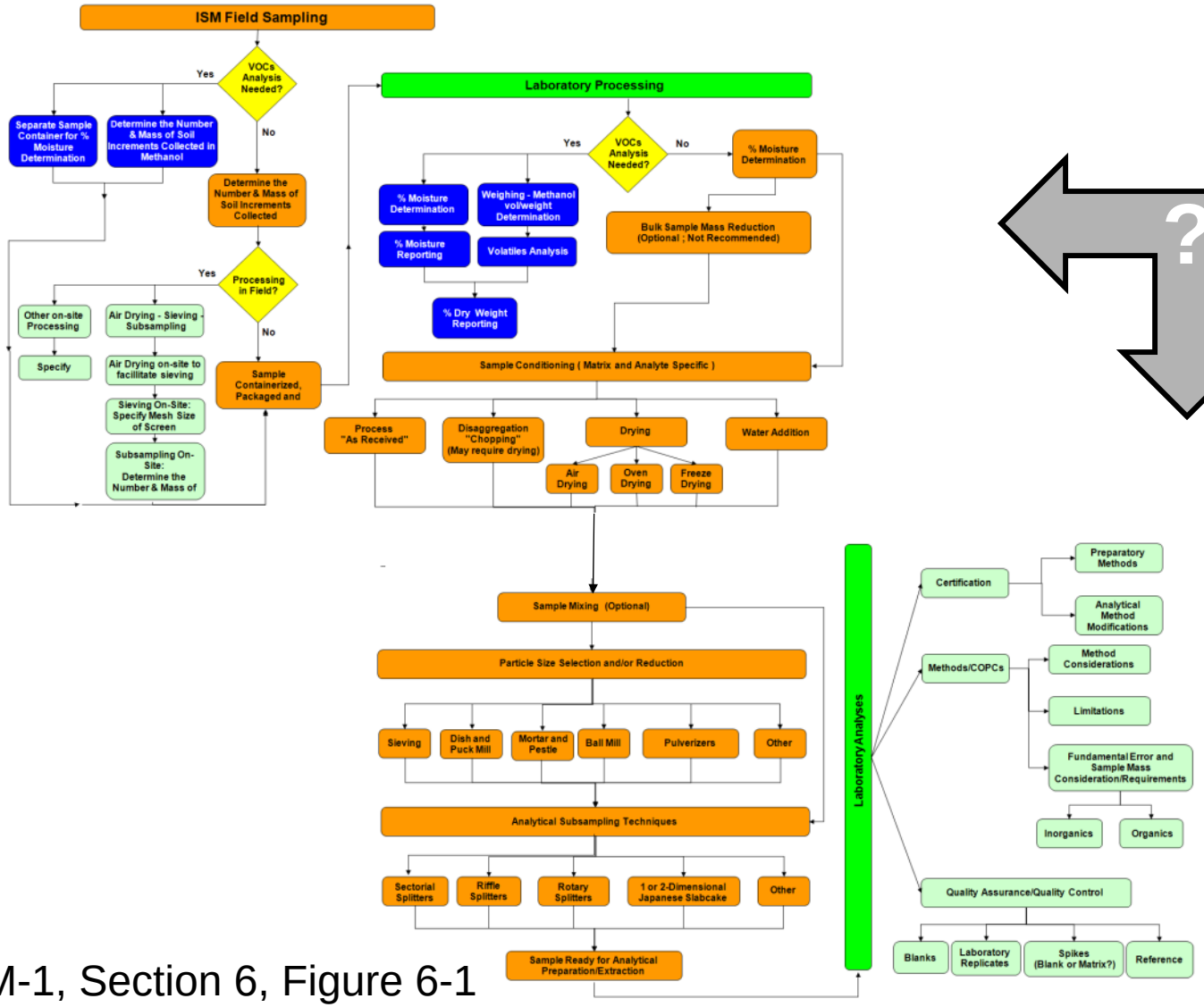
13. SUPPLEMENTARY NOTES
Presented at the 9th Annual DoD Environmental Monitoring and Data Quality (EDMQ) Workshop Held 26-29 March 2012 in La Jolla, CA. U.S. Government or Federal Rights License

14. ABSTRACT

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT **unclassified**	b. ABSTRACT **unclassified**	c. THIS PAGE **unclassified**	**Same as Report (SAR)**	**33**	

Real Life ISM has Choices

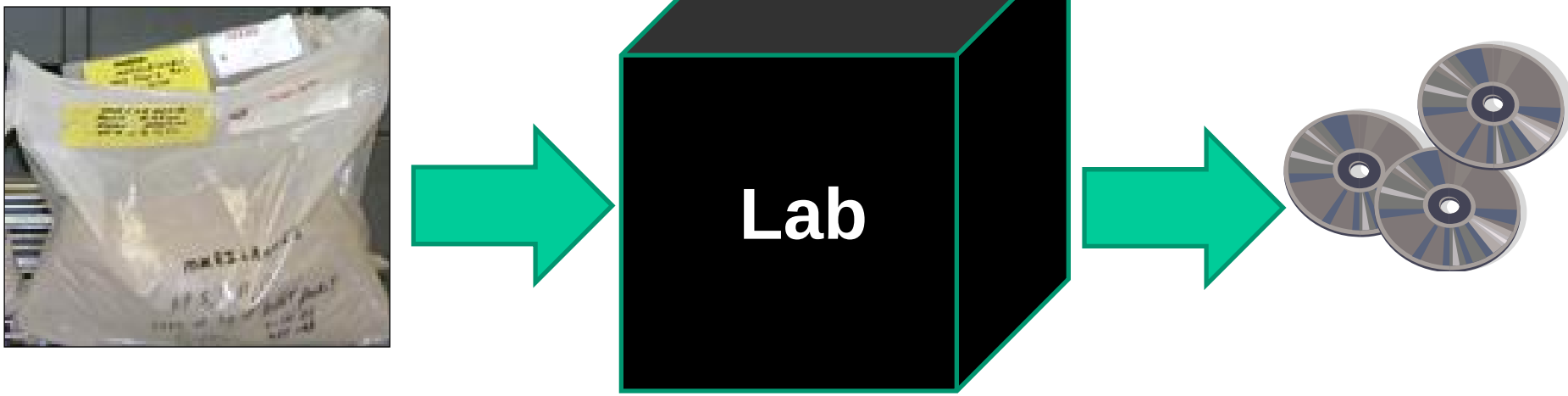


ITRC, ISM-1, Section 6, Figure 6-1

Analyte-Matrix Driven Options

- Pick the right option
  - More representative subsamples
  - Better precision
- Pick the wrong option
  - Poor and unknown bias

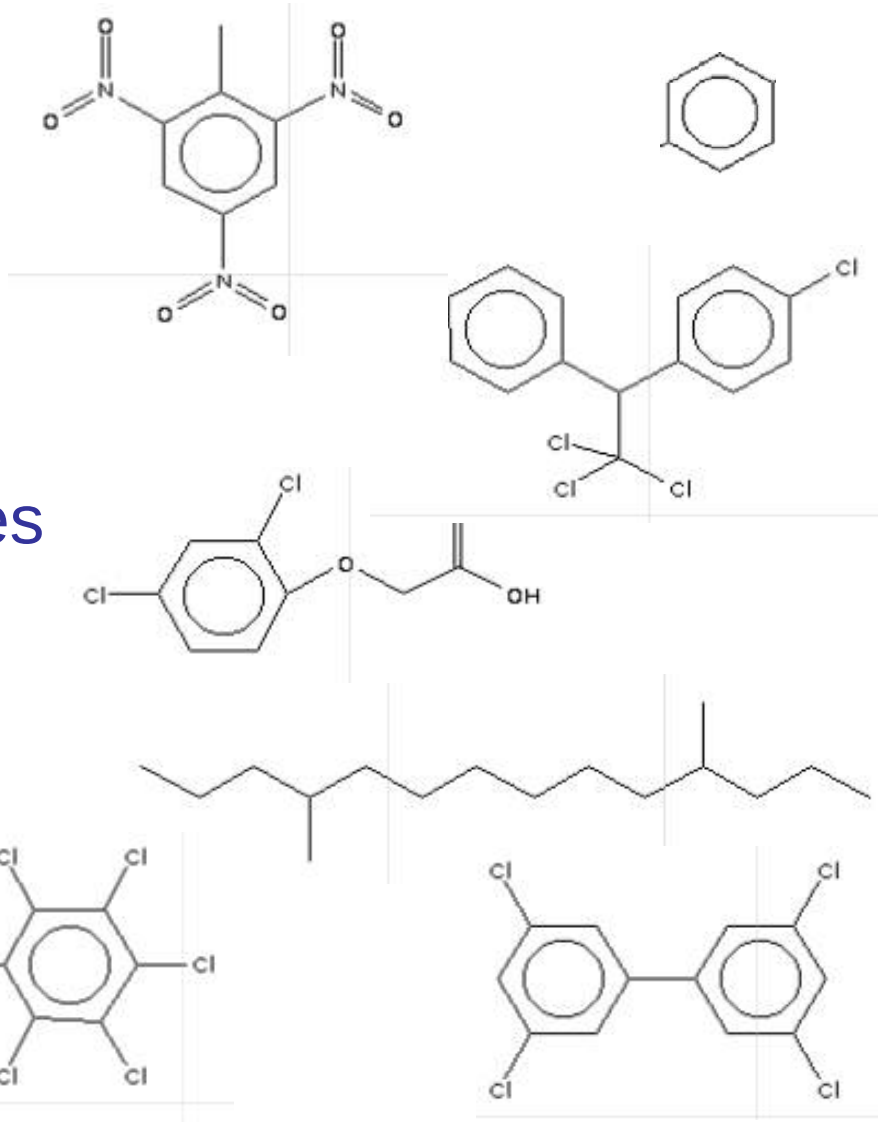
Include Lab Processing in Project Planning



ITRC, ISM-1, Section 6.1.1

Define the Analytes

- Volatile organics
- Energetics
- Metals, Hg
- PCBs
- Organochlorine pesticides
- Phenoxy acid herbicides
- Petroleum hydrocarbons
- Semivolatile organics
- Other



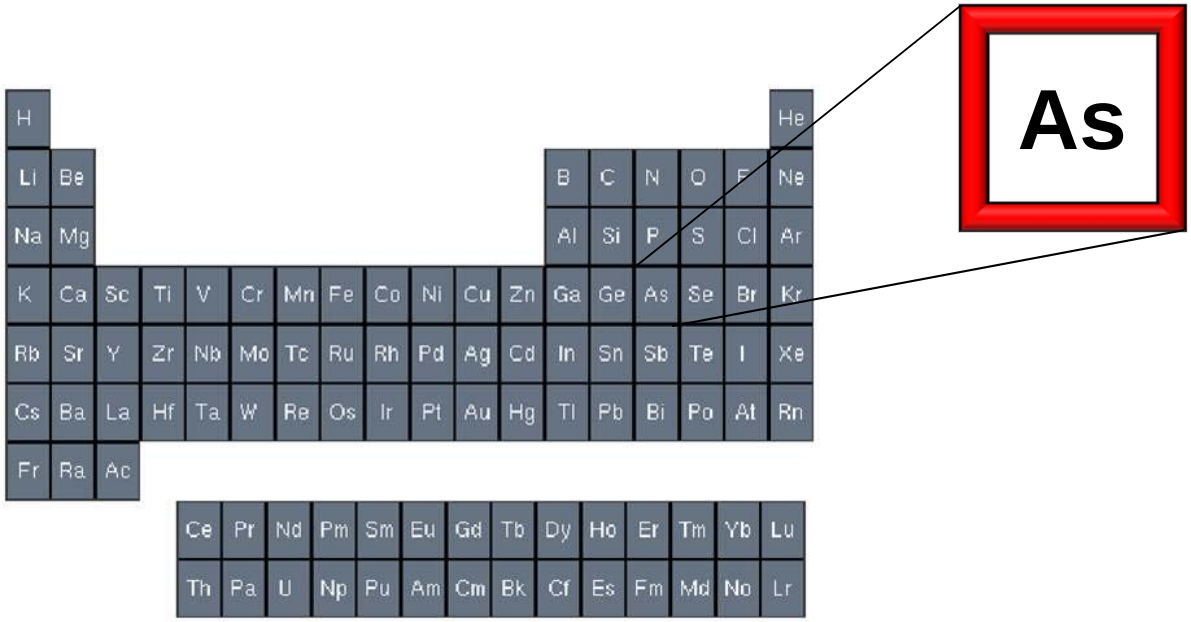
Coordinate VOC Sampling & Analysis

- Use methanol preservation
  - Methanol transport
  - Bottle sizes (large, medium, small)
- Analytical sensitivity limitations
  - Higher reporting limits
  - Selected Ion Monitoring GC-MS
    - Short analyte lists

ITRC, ISM-1, Section 6.2.1

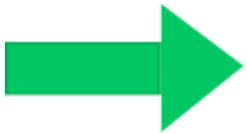
Florida Case Study: Contaminant of Concern

- Arsenic
  - From liquid applied pesticides



Periodic Table of Elements

Symbol Key



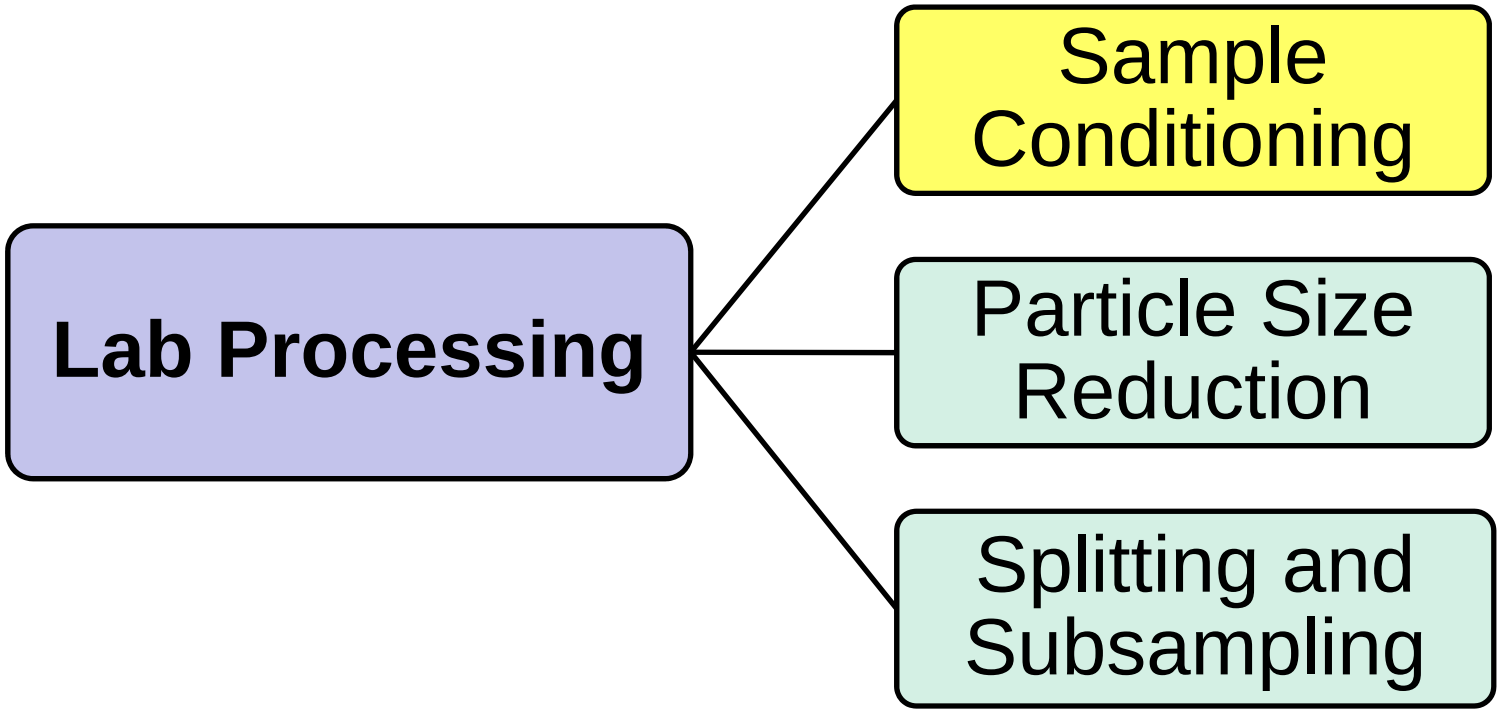
- Good effect

- Bad effect

- Result or statistic gets larger in value

- Result or statistic gets smaller in value

Lab Processing Roadmap



Condition the Sample

- Air drying
  - Room temperature – most common
  - Ventilation hood
  - Goal: Crushable agglomerates
  - Consider volatilization losses
    - Boiling point
    - Binding to soil particles
    - Potential for Loss Table
      - – Naphthalene
      - – Acenaphthene
      - – Benzo[a]pyrene

- Use other options when drying not appropriate

ITRC, ISM-1, Section 6.2.2.3

Florida Case Study: Air Drying Samples

- Arsenic
  - High boiling arsenic species
  - Volatilization loss not expected

Define Terms: Grinding

- Generic term for soil disaggregation or milling
- The grinding type or equipment must be specified to select a particular laboratory process

Define Terms: Disaggregating

- Breaking all the soil clumps into individual small particles, but keeping the small pebbles and hard crystalline particles intact

ITRC, ISM-1, Section 6.2.2.3

Define Terms: Milling

- Complete particle size reduction of all soil components including hard crystalline materials to a defined maximum particle size (e.g. < 75 µm)

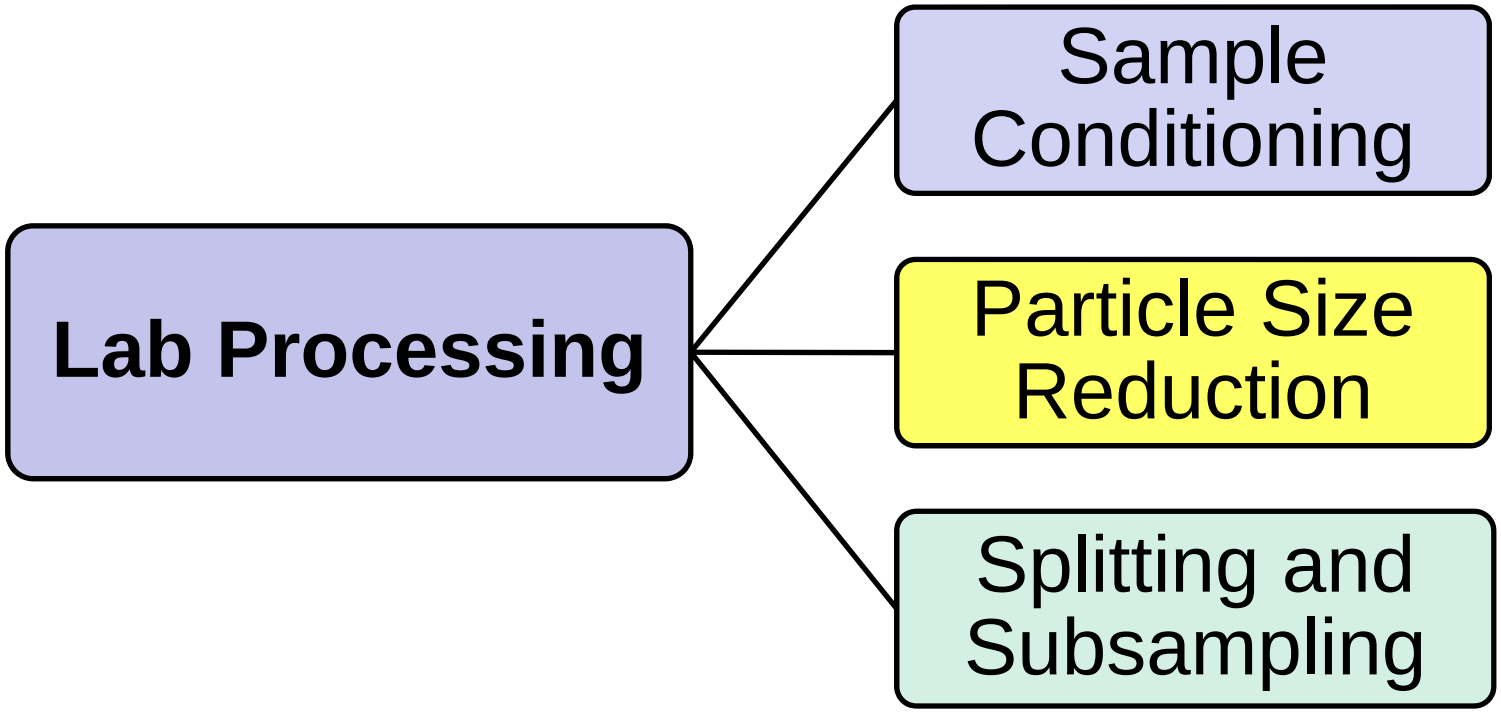
Picture from USACE-Alan Hewitt

ITRC, ISM-1, Section 6.2.2.5

Florida Case Study: Particle Size Reduction

- Disaggregation and sieving
  - Nugget effect expected to be small
    - Contaminant exposure sprayed as a liquid
- Mill
  - Puck mill
- Comparison study planned

Lab Processing Roadmap



To Mill or Not to Mill? (Particle Size Reduction)

- Recommended
  - Crystalline particles, fibrous threads, paint chips
  - Energetics, metals
- Strengths
  - Reduces variability
  - Reduces subsampling error
  - Facilitates mixing
  - Improves precision

Picture from USACE-Alan Hewitt

ITRC, ISM-1, Section 6.2.2.5

To Mill or Not to Mill

- Not recommended
  - Volatile, thermally labile, increased "availability"
  - Examples
    - Monochloro PCBs, reactive SVOCs, decane, elemental mercury
  - Limitations
    - Analyte losses
    - Metals contamination
    - Potential high bias to metals risk assessment (pebbles)

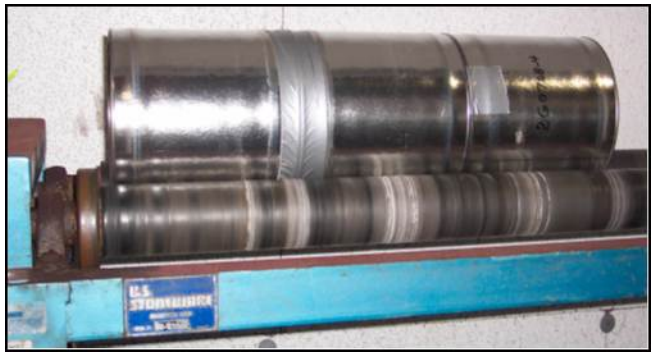
If uncertain, do milled & unmilled

ITRC, ISM-1, Section 6.2.2.5

How Best to Mill

- Puck mill or ring and puck mill
  - "Stable" energetics
- Ball mill
- Mortar and pestle
- Consider
  - Analytes
  - Concentration of interest
  - Mill materials
  - Particle size needed

Picture from USACE-Alan Hewitt

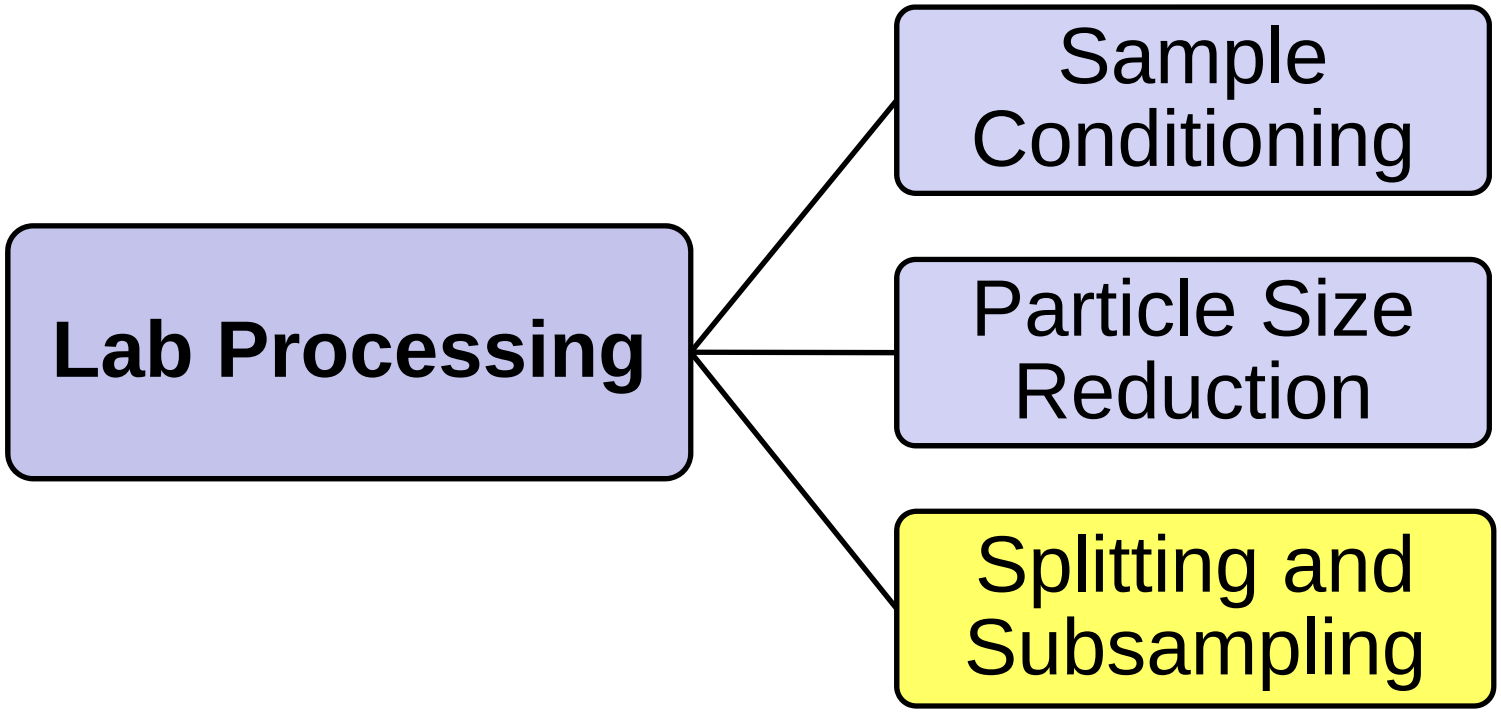


Example mills, other types are possible as well

Florida Case Study: Results Confirm Milling Not Needed

- Disaggregation and sieving
  - Nugget effect expected to be small
    - Contaminant exposure sprayed as a liquid
- Mill
  - Puck mill
- Results confirm milling not needed for this part of site
  - Small precision improvement with milling
  - No change in mean concentration

Lab Processing Roadmap



Dry Splitting Options

- Rotary sectorial splitter

ITRC, ISM-1, Section 6.2.2.7

Subsampling Options

- 2-Dimensional Japanese Slabcake

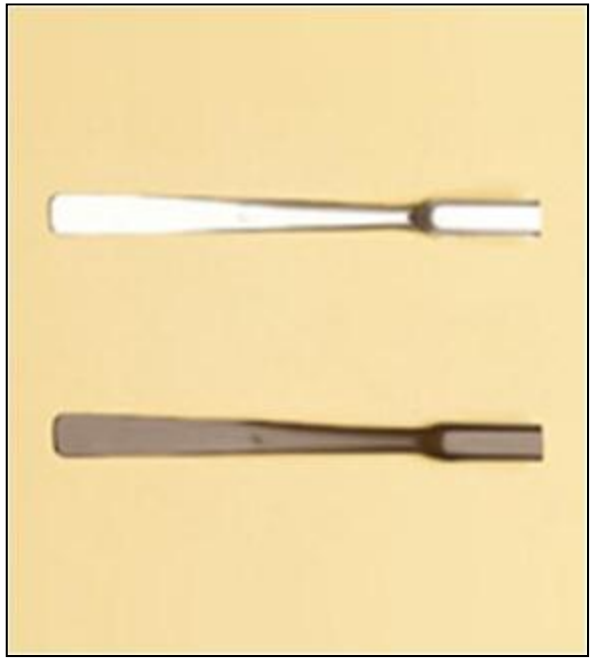
Dry

Wet

ITRC, ISM-1, Section 6.2.2.7

Subsampling Tools

- Square straight-sided scoops for dry non-cohesive soil

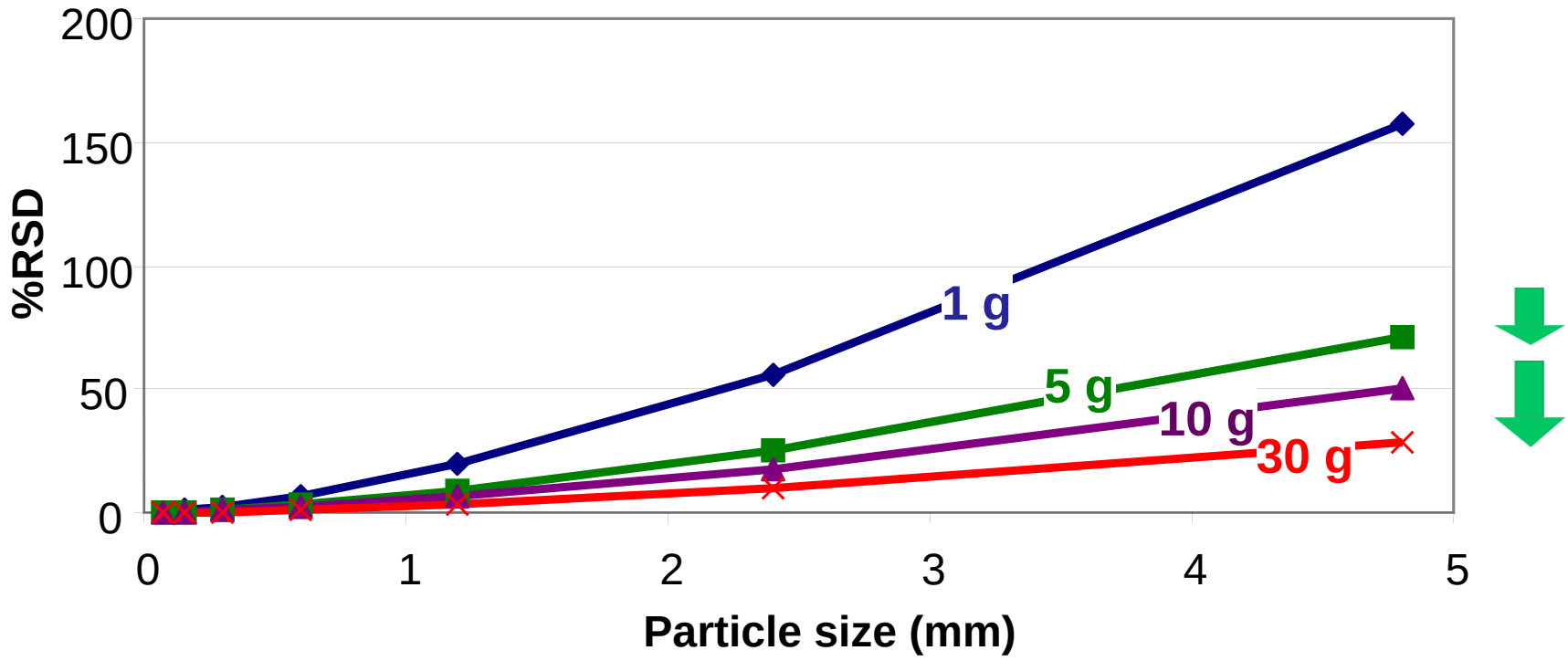


Florida Case Study: Choose Subsampling Process

- 2-D Slabcake Subsampling
  - Lower cost than sectorial splitter
  - More representative than “dig a spot”

Why Use Large Subsamples?

- Larger particles
  - Produce larger errors or require larger subsamples



ITRC, ISM-1, Section 6.3.3

Florida Case Study:
Nugget Effect Minimal

- 2 g subsamples on disaggregated aliquots
- 2 g subsamples on milled aliquots

- Low heterogeneity expected
  - Confirmed through replicates

Laboratory Quality Control Measures

- Laboratory equipment blanks
  - Limited clean matrices
- Laboratory control samples (LCS) and matrix spikes
  - Practicality of large scale spiking in kg samples
    - High cost
    - Limited availability
  - Introduced post ISM processing into subsample
- Subsampling replicates

Florida Case Study: Challenges with "Blank" Samples

- Ottawa sand method blank attempted for milling
  - Metals content of the sand was too variable
- Standard preparation batch QC
  - No laboratory control sample or matrix spike through ISM processes

Verify Laboratory Certification

- National Environmental Laboratory Accreditation Program (NELAP)

- Non-NELAP state accreditation

- Agency-specific accreditation
  - DoD Environmental Laboratory Approval Program

Cite Reference Methods

- Collecting and Processing of Representative Samples For Energetic Residues in Solid Matrices from Military Training Ranges
  - USEPA SW-846 Method 8330B, Appendix A
    http://www.epa.gov/osw/hazard/testmethods/pdfs/8330b.pdf
- Metals in Solid Matrices
  - USACE research effort
  - Planned SW-846 Method 3050 - Update V?

Use Alternate References

- ASTM D6323 Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities
  - ASTM 2003
- Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples
  - Gerlach 2003
- Laboratory Standard Operating Procedure

Lab Process "Big Rocks"